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From the Chemistry Lab to Licensing

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July 1998

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Space Administration

Lewis Research Center

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FROM THE CHEMISTRY LAB TO LICENSING*

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ABSTRACT

This is a story of technology maturation and transfer, and licensing. It traces the history of the patented ion-exchange material (IEM) from the accidental discovery that this polymer, United States Patent number 5,371,110, a battery separator of marginal performance, picked up copper from distilled water passing through corroded copper tubing in the laboratory, to a point where four organizations were awarded licenses to manufacture and market it and two other licenses are still pending. This story discusses in detail the problems of converting an immature technology into a mature and eventually commercialized technology, without dedicated resources. Readers will develop an appreciation for how the obstacles to maturation and licensing of the technology were faced and overcome. The lessons learned will be discussed, with the hope of enhancing the technology transfer process.

INTRODUCTION

The patented ion-exchange material (IEM) is a spinoff from space battery research conducted by Dr. Warren H. Philipp around 1987. During the course of his research he discovered, quite by accident, that the IEM was removing copper from the distilled water. A piece of IEM film turned blue when it was washed with distilled water. Apparently, the water had picked up the copper from a corroded line. Because of his serendipitous finding, he decided to investigate the film’s effect on other metal cations. He discovered that the IEM would extract other metal cations from contaminated water as well. (The IEM had marginal value as a batter separator.) As a result, the NASA Lewis Technology Transfer Office, the group responsible for transferring Lewis technology, made some early attempts to commercialize this technology but had little success.

Interest in the IEM was renewed in early 1991 when a CAMP (Cleveland Area Manufacturing Program) representative asked the NASA Lewis Technology Utilization Office if a technology existed that could be used by the electroplating industry to remove heavy metal cations from the rinse waters of plating operations such as a cadmium plating line. In response, the Technology Utilization Office mentioned the existence of the IEM technology but emphasized that the technology was immature. It would not soon be commercially available because considerable additional development was still required to raise its level of maturity.

In August 1991, Dr. Joseph Savino joined the NASA Lewis Technology Utilization Office. He took an optimistic view of this technology, which led him to further investigate whether the IEM might be a commercially viable technology, and if it was, to lead the effort that would result in the maturation of the IEM technology and its subsequent commercialization.

*This report is an updated version of the original paper submitted in March, 1996 for presentation at the Technology Transfer Conference, July 1996 and subsequently published in the conference proceedings (see supplementary notes). This recent version includes the major developments that occurred between Mach 1996 and June, 1998.

†Joseph M. Savino was a technology transfer engineer with Advanced Manufacturing Center, an affiliate of the Cleveland Advanced Manufacturing Program (CAMP) and the facilitator of this project. Previously he was a NASA Lewis Research for 34 years.

‡ Kenneth W. Street, a coinventor of the IEM, is a research chemist in the NASA Lewis Surface Science Branch. He has published nearly 100 journal papers, articles, and technical presentations.

§ Warren H. Philipp, a coinventor of the IEM, is the president of Philipp Scientific, Inc., Olmsted Falls, OH. Previously he was a research chemist in the Ceramics Branch at NASA Lewis for 33 years.

EARLY ASSESSMENT OF THE COMMERCIAL POTENTIAL

Having no funding available to conduct a market-potential study and knowing there were already a number of ion-exchange resins on the market, Drs. Philipp and Savino decided to consult wastewater experts and organizations that would know if there was a need for such a product in the marketplace. The opinion of a Cleveland State University expert in wastewater treatment was that the IEM was an "interesting material" which merited further development and evaluation. Representatives of the North East Ohio Regional Sewer District thought that the IEM would make a good polishing agent; that is, it could be used after the bulk of heavy metals had been removed from the water, to further remove such metals before discharging the water into the environment. Discussions with some Cleveland-based companies revealed that commercially available ion-exchange resins which were effective in calcium-laden water were very expensive—in the range of $200 to $2000 per cubic foot. These facts and opinions, plus the knowledge that the NASA IEM had very good cation-uptake properties and was rechargeable, led Drs. Philipp and Savino to conclude that it was worthwhile to conduct more research and development (R&D) in an effort to advance the maturity of the IEM technology—a necessary step toward commercialization.

In mid-1991, Dr. Philipp teamed with Dr. Kenneth Street, then at the NASA Lewis Office of Environmental Programs in an effort to further develop the IEM technology. Because of his knowledge and experience with environmental pollution problems, Dr. Street fully appreciated the significance of Dr. Philipp's work and the need for greater resources (than were generally available at Lewis) without which advancement of the technology would be nearly impossible. Drs. Street and Philipp applied for and were awarded a grant from the Lewis Director's Discretionary Fund to do the needed R&D. This initial infusion of funds enabled Drs. Philipp and Street to make significant improvements in the methods for making the IEM. They were also able to characterize the rate and capacity of its up-take properties for a number of heavy metals, including lanthanides as stand-ins for some radioactive elements. At that time it was deemed advantageous to develop methods for making the IEM in a variety of forms, such as beads and granules (for use in packed columns—a standard method for extracting cations from contaminated water), powder (for use in batch processes), and filaments (for use in fiber-wound filters that also act as ion exchangers). However, for convenience, a decision was made to use the IEM in the form of a thin film because it could be made very easily in the laboratory. A second area of research was inititated to optimize the formulation and processing for industrial production.

RESOURCES FOR TECHNOLOGY MATURATION AND COMMERCIALIZATION

It was not clear in 1991 if the IEM technology would be of interest to U.S. industry. There were many features and characteristics of the IEM that were not yet known but that were considered important; thus, further research was required. For the IEM to be a viable technology, the following information was needed:

(1) Methods for making the IEM into beads, granules, thin films, powder, and fibers on a commercial scale
(2) The IEM's uptake and capacity properties for many toxic heavy metals in a variety of media
(3) The recycling service life of the IEM
(4) Methods for disposing of toxic-metal-laden IEM
(5) Product costs

To meet these needs, the following goals were set and the accompanying approaches were used.

Technology Maturization Goals and Approaches

(1) **Goal:** Develop methods for making IEM film, beads, granules, and powder on a commercial scale.

Approach: First we developed all four of these IEM forms either in the Lewis chemistry lab or outside Lewis in small-scale equipment such as spray dryers. The thin film form was easy to make in the lab; the beads, granules, and powder were made by the Southwest Research Institute, San Antonio, TX, under a grant. Southwest Research Institute is a nonprofit center of excellence in the technology of microencapsulation. Having proven that all these forms could be made on the small scale, we proceeded to contract out the efforts to make the various forms to companies that manufacture similar products.

(2) **Goal:** Measure the working pH range, uptake rate, and uptake capacity of IEM for a wide variety of heavy metal cations, such as lead, zinc, cadmium, mercury, and many others.

Approach: Measure these properties in the NASA Lewis Research Center's chemistry labs by using chemists and lab technicians brought in, as needed, under grants, or hired, as funding permitted, through a support service contractor.

(3) **Goal:** Acquire performance data and experience in at least one real application.

Approach: Enter into joint projects with a variety of industrial and government organizations interested enough in the IEM technology to participate in testing and evaluating the IEM in their particular applications. Among the applications were the removal of (a) lead from maple syrup, (b) lead from drinking water and sea water, (c) zinc from the rinse water of a zinc electroplating process, and (d) lead from a soil washing process.

(4) **Goal:** Patent the IEM technology for the benefit of the U.S. economy.

Approach: Use the Lewis patent attorneys to prepare and submit all the paper work required by the U. S. Patent Office. The patent on the IEM formulation was granted to Drs. Philipp and Street on December 6, 1994.

Commercialization Goals and Approaches

(1) **Goal:** Publicize the existence of and disseminate data and information about the IEM.

Approach: There were several ways at our disposal for publicizing the IEM and what it could do. One of the most effective ways was via press releases to the print media. These releases were prepared by writers in the Lewis Research Center's Media Relations Office and sent to numerous newspapers, magazines, and trade journals. The articles generated hundreds of telephone inquires from which our most interested companies emerged. A second method to publicize the new technology was to make presentations at technology transfer conferences and expositions and to individual organizations such as the U.S. Environmental Protection Agency (EPA) and the U.S. Army Corps of Engineers. The third method was to provide a handout called *TECH FOCUS* to interested parties who inquired. This handout contained a description of the IEM and some of its uptake performance data. The *TECH FOCUS* has been updated from time to time to include new information as it was developed. It is currently in its third revision.

(2) **Goal**: Identify potential users and manufacturers of the material.

Approach: We depended on receiving responses to the news releases, presentations, and technology expositions to identify potential users and manufacturers. We also expected that the NASA regional technology centers around the country would occasionally refer interested companies to us.

(3) **Goal:** Determine economic feasibility of the IEM.

Approach: Cost effectiveness was one of the most difficult features to determine without resources with which to conduct a cost analysis. The course we took was to ask companies what they paid for comparable ion-exchange resins (usually we were told $200 to $2000 per cubic foot). Then we asked those companies interested in manufacturing and marketing the IEM if their cost analysis showed it to be cost effective; they said it was. We contracted, in summer 1995, to two companies to develop manufacturing processes for making beads and granules. They were asked to determine a firm cost per pound f.o.b. at which they would sell the granules and beads. Comparing this figure with the market price for available ion-exchange resins would tell us whether the IEM granules and beads were cost-effective ion-exchange resins. Other parameters which influence cost effectiveness like reduction in floor space required and cost per unit of water treated were considered to be beyond the scope of this program. In 1996, a cost-of-materials was made. It was estimated that the materials alone would cost about $2.50 per pound. Assuming that the cost to manufacture would add $1.50 per pound,[1] the total cost to manufacture this material would be under $4.00 per pound product (around $120 per cubic foot).

[1]The $1.50 per pound estimate was provided by an existing polymer manufacturer.

(4) **Goal:** Identify and encourage potential licensees to participate in joint test and evaluation projects.

Approach: Potential licensees are generally companies that are forward-looking and interested in staying ahead of their competition (as are nonprofit institutions). When such companies contacted us (in response to one of our press releases, presentations, etc.), our approach was to educate them about the IEM and its potential benefits. Once a company decided to work with us, a nonreimbursable Space Act Agreement (SAA) between the company and NASA was signed. The SAA committed both parties to work together to evaluate the performance of the IEM in the company's application. This form of cost sharing allowed further technology development without the need for funding by the NASA investigators. Not all companies that applied for a license thought it was necessary to work with NASA; they were convinced the IEM would improve their bottom line without engaging in a joint project.

(5) **Goal:** License the technology to at least one company.

Approach: Potential licensees are the aforementioned companies, namely those with whom NASA had a Space Act Agreement. If during the joint project a company became convinced the IEM would be of value, they applied for a license for their specific application. We encouraged companies with whom NASA did not have an SAA to apply for a license as well.

To meet our goals, we judged we would need the following:

(1) A well-equipped chemical laboratory
(2) Chemists and chemical technicians
(3) Services to publicize and disseminate data and information
(4) Industrial partners for joint test and evaluation projects leading to commercialization
(5) Methods for judging the potential cost effectiveness
(6) Methods for identifying suitable applications and potential licensees
(7) Legal services for writing cooperative agreements, preparing patent applications, and preparing licensing agreements

NASA had the chemical laboratory, the services to publicize and disseminate data and information, the legal services, and some, but not enough, chemists and technicians, and methods for identifying applications and licensees. At the ouset, there were no industrial partners, nor methods for judging cost effectiveness or for identifying suitable applications. Nor was there any funding with which to conduct cost analyses and market assessments. The resources that were not available clearly had to be acquired, by a means not readily apparent to us at the time, or the development and commercialization of the IEM would be significantly delayed or not happen at all.

How the Resources Were Acquired and Used

The primary resource that jump-started the IEM technology maturation was funding with which to hire the needed extra chemists and chemical technicians. Although funding is often difficult to find because of fierce competition for the same few dollars, in this case the potential of the IEM convinced the funding sources to support this project. Funding was obtained from (1) the Lewis Director's Discretionary Fund ($154,000), (2) the U.S. EPA ($75,000), (3) the U.S. Army Corps of Engineers ($60,000), (4) NASA headquarters ($220,000), and (5) the Lewis Technology Utilization Office ($210,000). The total funding from 1991 through 1995 amounted to about $700,000, not including the salaries of the authors and participating civil servants such as the patent attorneys and writers in the Media Relations Office.

The funds were used to award grants and contracts to

- Local college chemistry professors, their students, professors of chemical engineering, and chemical technicians. These professionals were employed primarily to carry out the many chemistry experiments and analyses involved in determining the uptake characteristics of the IEM for a wide range of toxic-metal cations in a variety of different media ranging from tap water to maple syrup.
- The Southwest Research Institute, San Antonio, TX, to develop methods for making the IEM into powder, coated magnetic particles, and granules and beads.

• Chemsultants International, Mentor, OH, to develop production methods for making IEM thin sheets.

• Two companies, Pore Technology Ltd., Framingham, MA, and Howard Industries, Inc., Columbus, OH, who were contracted to develop the processes with which to make the beads and granules needed for high volume production.

The Lewis institutional resources were used to

• Publicize the existence of the IEM and its characteristics.

• Produce a handout containing a description of the IEM and its uptake properties for a wide range of toxic metals. These were given to anyone requesting information.

• Draw up Space Act Agreements between interested companies and NASA, whereby the parties agreed to jointly test and evaluate the IEM with respect to the companies' applications.

• Draft and submit the paperwork required by the patent application process.

• License the patented IEM.

The publicity given the IEM in news articles and the presentations at conferences attracted the attention of a great many companies and public institutions. From this group of organizations, there emerged potential licensees as well as a number of other interested persons. In effect, the news releases, articles, and presentations served as the IEM marketing and market-potential activities. The results show this form of publicity worked very well; it was slow but very effective. The truly interested companies and licensees were identified.

IEM TECHNOLOGY MATURATION

In the time period from 1991 to the present, the following notable advances have been made in the IEM technology:

• Methods have been developed by which the various forms (powder, beads, thin films, granules, and fibers) can be made on a laboratory scale. Small scale production runs for powder, beads, and granules were demonstrated by contracting to a nonprofit research institution. A preliminary production method for making a fiberglass-reinforced thin film has been demonstrated: five rolls of sheets 1 ft wide by 1000 ft long were produced.

• Since March 1996, further improvements were made in the commercial production process(es) for making the IEM in the form of granules. This work started in support of a demonstration project that showed the IEM could be used to remove lead from soil washings at a rifle range.

• Uptake rates for approximately 20 metal cations have been measured by using the film, bead, granule, and powder forms.

• The capacities of the various forms of the IEM were determined indirectly, and then were experimentally confirmed for zinc.

• The mechanism for IEM binding to metals was confirmed by infrared studies by the research group led by Dr. Joseph Gorse of the chemistry department at Baldwin-Wallace College.

• The IEM was successfully used to coat magnetic powder particles.

• A patent for the compositions and application of the IEM was granted to Drs. Warren Philipp and Kenneth Street in December 1994.

• With funding from the U.S. Army Corps of Engineers, Dr. Street and a university colleague, Dr. S.P. Tanner of the University of West Florida, developed a user-friendly test kit for determining if the lead in drinking water is above the EPA recommended level (a similar copper test kit was independently developed).

• With funding support from the EPA, Dr. Street and his team of chemists have demonstrated that the IEM can remove lead from the leachates used to extract lead from soil contaminated with lead paint and from soil washed at a rifle range.

• In cooperation with the states of Vermont and New Hampshire, we showed that the IEM can remove almost all the lead found in maple syrup.

• A method and apparatus for processing industrial wash water using moving film was developed, characterized, and demonstrated in a zinc electroplating line. This was done in collaboration with Drs. Orhan Talu and D.B. Shah of the Department of Chemical Engineering at Cleveland State University.

• The IEM has been termed cost-effective by one of the potential licensees. This company produces more than 100 chemical formulations for the wastewater treatment industry. (The company intended to apply for a license but has not done so because it has been purchased by another company.)

• In collaboration with Dr. Manny Uy of the Johns Hopkins University Applied Physics Laboratory, we demonstrated that the IEM is effective in removing lead from sea water, a military application.

IEM LICENSING

At the time (in the Spring 1995) of this writing, five organizations and one individual have applied for licenses to make and/or use the IEM. The applications range from lead removal from drinking water and maple syrup to the removal of many toxic metals from various kinds of industrial wastewaters. By the fall of 1997 licenses have been granted for various applications and manufacturing of the IEM. One company, Howard Industries, Inc., Columbus, Ohio, has produced experimental lots of IEM for commerical application and is engaged in making and marketing the IEM. The second licensee, Solar Universal, Inc., Cleveland, Ohio is assembling a manufacturing and marketing team. Two other licenses were granted, but those companies have not pursued commercialization of the IEM.

SUMMARY COMMENTS

The maturity of the IEM technology has been significantly advanced beyond what it was when discovered in the laboratory. It is a versatile and promising ion-exchange polymer, but it is not yet a fully mature technology. Nevertheless, significant progress has been made; bringing the technology to this highly mature state has encouraged a number of organizations to apply for licenses. The hundreds of inquiries received, mostly from end users, indicate there is considerable interest in the IEM in the commercial world, and therefore, a sizable market for the product.

When the IEM is actually being produced on a routine basis and used by one or more organizations, true technology transfer and true commercialization will have occurred; this is the ultimate metric. Licensing a technology is a critical step and a significant indicator along the road to true commercialization. In the case of the IEM, commercialization appears to be only a small step away. At the time of this writing (March, 1998), Howard Industries, a commerical licensee, has spun off a subdivision to manufacture and distribute the IEM. Once the final formulation and manufacturing process, i.e. production line, are established and continuous production begins, the IEM will be a success story. The other licensee, Solar Universal., is assembling a manufacturing and marketing team.

The licensees will have to complete the process of bringing the IEM to the commercial production and application states. When this happens, it will be a clear indication of the value and cost effectiveness of the IEM as a new and better ion-exchange resin.

The publicity given the IEM in news articles and the presentations at conferences attracted the attention of a great many companies and public institutions. From this group of organizations, the potential licensees and interested persons emerged. In effect, the news releases, articles, and presentations served as the IEM marketing activity and means of evaluating market potential. Judging from the results, we think this form of publicity worked very well; the truly interested companies and licensees were identified.

FUTURE IMPACT

The long-term impact of the IEM technology will not become apparent for some time, since we expect small entrepreneurial ventures to continue emerging as new applications are found for the novel properties of this new material. For example, ion-exchange materials have never been practically incorporated into environmental monitoring or clinical sensors. However, because of the exceptional transparency of this material, one university researcher completed tests indicating the viability of incorporating the IEM into these sensors. As a second example, a large number of applications for ion-exchange films have been presented in the literature, but they have not been commercialized because of problems associated with past film ion exchangers. With the development of the NASA IEM, many of these problems have been eliminated, so ultimately this application may lead to the greatest success of the material.

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13. ABSTRACT ***(Maximum 200 words)***

This is a story of technology maturation and transfer, and licensing. It traces the history of the recently patented ion-exchange material (IEM) from the accidental discovery that this polymer, a battery separator of marginal performance, picked up copper from distilled water passing through corroded copper tubing in the laboratory, to a point where five organizations and one individual have applied for licenses to manufacture and market it or to use it in a wide variety of applications. This story discusses in detail the problems of converting an immature technology into a mature and eventually commercialized technology, without dedicated resources. Readers will develop an appreciation for how the obstacles to maturation and licensing of the technology were faced and overcome. The lessons learned will be discussed, with the hope of enhancing the technology transfer process.

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